

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
27 September 2001 (27.09.2001)

(10) International Publication Number  
**WO 01/70926 A1**

(51) International Patent Classification<sup>7</sup>: C11D 17/00, 1/94, 3/20, 17/04, A61K 7/50, A45D 40/00

(21) International Application Number: PCT/EP01/02458

(22) International Filing Date: 5 March 2001 (05.03.2001)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:  
60/190,624 20 March 2000 (20.03.2000) US

(71) Applicant (for AE, AG, AU, BB, BZ, CA, CY, GB, GD, GH, GM, IE, IL, KE, LC, LK, LS, MN, MW, NZ, SD, SG, SL, SZ, TT, TZ, UG, ZA, ZW only): UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB).

(71) Applicant (for AL, AM, AT, AZ, BA, BE, BF, BG, BJ, BR, BY, CF, CG, CH, CI, CM, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, FR, GA, GE, GN, GR, GW, HR, HU, ID, IS, IT, JP, KG, KP, KR, KZ, LR, LT, LU, LV, MA, MC, MD, MG, MK, ML, MR, MX, MZ, NE, NL, NO, PL, PT, RO, RU, SE, SI, SK, SN, TD, TG, TJ, TM, TR, UA, UZ, VN, YU only): UNILEVER NV [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL).

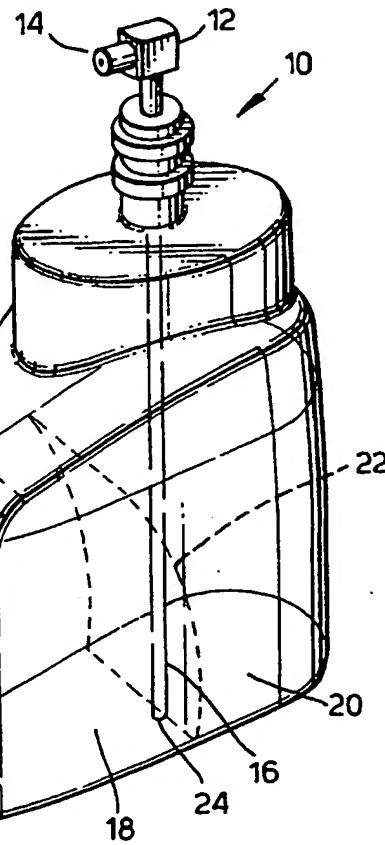
(71) Applicant (for IN only): HINDUSTAN LEVER LIMITED [IN/IN]; Hindustan Lever House, 165/166 Backbay Reclamation, Mumbai 400 020, Maharashtra (IN).

(72) Inventor: MITRA, Shuman; 5608 Croydon Avenue, San Jose, CA 95118 (US).

(74) Agents: ROTHS, Maria, Johanna, Francisca et al.; Unilever PLC, Patent Dept., Colworth House, Sharnbrook, Bedford, Bedfordshire MK44 1LQ (GB).

[Continued on next page]

(54) Title: EXTRUDABLE MULTIPHASE COMPOSITION COMPRISING LAMELLAR PHASE INDUCING STRUCTURANT IN EACH PHASE



(57) Abstract: The invention relates to a plurality of liquid cleansing compositions in lamellar phase which possess a lotion-like appearance conveying perceptions of enhanced moisturization contained in a partitionless container. This multiphase composition is stable upon storage and is dispensed as a striped product where typically one stripe has a cleansing function and a second stripe has a moisturising function.

**WO 01/70926 A1**



(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW.

IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

**Published:**

- *with international search report*
- *before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments*

(84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE,

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

- 1 -

EXTRUDABLE MULTIPHASE COMPOSITION COMPRISING LAMELLAR PHASE  
INDUCING STRUCTURANT IN EACH PHASE

5 The present invention relates to extrudable multiphase liquid cleansing compositions of the type typically used in skin cleansing or shower gel compositions which compositions are "structured" lamellar phase compositions.

10 Dual Phase Cleansing and Related Compositions:

Compositions which both provide a cleansing function and a moisturizing benefit are known. For example, WO 90/13283 to Green, et al., incorporated herein by reference, discloses 15 compositions comprising an acyl ester of an isethionic acid salt, a long chain fatty acid, a moisturizer component and optional soap.

One problem which had been previously encountered with such 20 dual purpose compositions is that they either contain an insufficient level of moisturizer component; or an insufficient amount deposits on use.

Another problem associated with such dual cleansing and 25 moisturizing compositions is instability. According to WO 94/03152 to Helliwell, included herein by reference, which is concerned with shower gels comprising a non-soap detergent, silicone oil and cationic polymers, the maximum average droplet size of the silicone oil that can be used is 30 2 microns, if product stability is to be maintained.

- 2 -

In applicants U.S. Pat. No. 5,612,307 issued to Chambers, et al., included herein by reference, it was found that enhanced deposition of benefit agent could be obtained in a stable formulation by using a dual cleansing and 5 moisturizing product in which the cleansing and moisturizing components were separately, but combinedly dispensed from a packaging means as discrete domains/stripes.

More specifically, the compositions of Chambers, et al. 10 comprised a surfactant-containing base formulation and a benefit agent wherein the benefit agent and base formulation were physically separate (not in direct contact) but were nonetheless dispensable from a single packaging means comprising both the base formulation and benefit agent as 15 individual stripes. The stripes had width of at least 1000 microns and base formulation and benefit agent stripes were not post mixed prior to use (compared to EP 468,703 to Unilever where post-mixing is required).

20 In applicants U.S. Pat. No. 5,929,019 issued to Puvvada et al., incorporated herein by reference, applicants modified the benefit agent stripe in the same separately dispensed, non-mixed prior to use, dual cleanser/moisturizer compositions described by Chambers, et al, to include 25 surfactant.

Multiphase cleansing and cosmetic compositions which are not segregated in their package are also known. For example, in 30 U.S. Pat. No. 5,059,414 issued to Dallal et al. incorporated herein by reference, a multi-phase high viscosity cosmetic product containing two or more independent products in a

- 3 -

single container, along with simultaneous dispensing, is described. However, this patent related to isotropic products for the hair, whereas the present invention relates to lamellar liquids for personal care (hand, body and hair).

5

WO 9824399 to Bordat *et al.*, included herein by reference, describes highly viscous, separate aqueous and oil phase emulsion compositions which are squeezed out together as a single strand from a tube dispenser for use with the skin, 10 body or hair. In comparison, the present invention uses lamellar liquids with low shear viscosity values between 80-300 K cps.

#### Lamellar Compositions:

15

The rheological behaviour of all surfactant solutions, including liquid cleansing solutions, is strongly dependent on the microstructure, i.e., the shape and concentration of micelles or other self-assembled structures in solution.

20

When there is sufficient surfactant to form micelles (concentrations above the critical micelle concentration or CMC), for example, spherical, cylindrical (rod-like) or discoidal micelles may form. As surfactant concentration increases, ordered liquid crystalline phases such as lamellar phase, hexagonal phase or cubic phase may form. The lamellar phase, for example, consists of alternating surfactant bilayers and water layers. These layers are not generally flat but fold to form submicron spherical onion like structures called vesicles or liposomes. The hexagonal phase, on the other hand, consists of long cylindrical 25 30

- 4 -

micelles arranged in a hexagonal lattice. In general, the microstructure of most personal care products consist of either spherical micelles; rod micelles; or a lamellar dispersion.

5

As noted above, micelles may be spherical or rod-like. Formulations having spherical micelles tend to have a low viscosity and exhibit Newtonian shear behaviour (i.e., viscosity stays constant as a function of shear rate; thus, if easy pouring of product is desired, the solution is less viscous and, as a consequence, it doesn't suspend as well). In these systems, the viscosity increases linearly with surfactant concentration.

10 15 Rod micellar solutions are more viscous because movement of the longer micelles is restricted. At a critical shear rate, the micelles align and the solution becomes shear thinning. Addition of salts increases the size of the rod micelles thereof increasing zero shear viscosity (i.e., viscosity when sitting in bottle) which helps suspend particles but also increases critical shear rate (point at which product becomes shear thinning; higher critical shear rates means product is more difficult to pour).

20 25 Lamellar dispersions differ from both spherical and rod-like micelles because they can have high zero shear viscosity (because of the close packed arrangement of constituent lamellar droplets), yet these solutions are very shear thinning (readily dispense on pouring). That is, the 30 solutions can become thinner than rod micellar solutions at moderate shear rates.

- 5 -

In formulating liquid cleansing compositions, therefore, there is the choice of using rod-micellar solutions (whose zero shear viscosity, e.g., suspending ability, is not very good and/or are not very shear thinning); or lamellar dispersions (with higher zero shear viscosity, e.g. better suspending, and yet are very shear thinning). Such lamellar compositions are characterized by high zero shear viscosity (good for suspending and/or structuring) while simultaneously being very shear thinning such that they readily dispense in pouring. Such compositions possess a "heaping", lotion-like appearance which convey signals of enhanced moisturization.

In order to form such lamellar compositions, however, some compromises have to be made. Firstly, generally higher amounts of surfactant are required to form the lamellar phase. Thus, it is often needed to add auxiliary surfactants and/or salts which are neither desirable nor needed. Secondly, only certain surfactants will form this phase and, therefore, the choice of surfactants is restricted.

In short, lamellar compositions are generally more desirable (especially for suspending emollient and for providing consumer aesthetics), but more expensive in that they generally require more surfactant and are more restricted in the range of surfactants that can be used.

When rod-micellar solutions are used, they also often require the use of external structurants to enhance

- 6 -

viscosity and to suspend particles (again, because they have lower zero shear viscosity than lamellar phase solutions). For this, carbomers and clays are often used. At higher shear rates (as in product dispensing, application of 5 product to body, or rubbing with hands), since the rod-micellar solutions are less shear thinning, the viscosity of the solution stays high and the product can be stringy and thick. Lamellar dispersion based products, having higher zero shear viscosity, can more readily suspend emollients 10 and are typically more creamy. Again, however, they are generally more expensive to make (e.g., they are restricted as to which surfactants can be used and often require greater concentration of surfactants).

15 In general, lamellar phase compositions are easy to identify by their characteristic focal conic shape and oily streak texture while hexagonal phase exhibits angular fan-like texture. In contrast, micellar phases are optically isotropic.

20 It should be understood that lamellar phases may be formed in a wide variety of surfactant systems using a wide variety of lamellar phase "inducers" as described, for example, in U.S. Pat. No. 5,952,286 issued to Puvvada, et al.

25 Generally, the transition from micelle to lamellar phase are functions of effective average area of headgroup of the surfactant, the length of the extended tail, and the volume of tail. Using branched surfactants or surfactants with smaller headgroups or bulky tails are also effective ways of 30 inducing transitions from rod micellar to lamellar.

- 7 -

One way of characterizing lamellar dispersions include measuring viscosity at low shear rate (using for example a Stress Rheometer) when additional inducer (e.g., oleic acid or isostearic acid) is used. At higher amounts of inducer,  
5 the low shear viscosity will significantly increase.

Another way of measuring lamellar dispersions is by the use of freeze fracture electron microscopy. Micrographs generally will show lamellar microstructure and close packed.  
10 organization of the lamellar droplets (generally in size range of about 2 microns).

Applicants have now surprisingly discovered that a stable, extrudable multiphase product may be prepared. The term  
15 multiphase product is defined herein as the combination of two or more distinct lamellar compositions having viscosities of at least about 80,000 cps (T-bar) at 25°C. Preferably the viscosity has an upper limit of 300,000 cps at 25°C in order to facilitate filling containers and  
20 dispensing with a conventional pump bottle.

The lamellar phases may have substantially the same or different compositions, but preferably the phases have similar rheological properties, such as viscosity, etc. The  
25 lamellar phases preferably have different colors or other visual differences and preferably are filled vertically or in a pulsating manner in a single container without any partitions, i.e. "partitionless".

30 Squeezing a flexible container holding the inventive product may dispense the product but a single pump, or the like, is

- 8 -

preferably used to dispense the product. When dispensed, each phase of the multiphase inventive product should be present in a concentration range of 1 - 99 weight %. In this manner, duality in the case of a two phase system, can be advantageously, economically, and visually communicated through a single, partitionless container.

Another advantage of the inventive product is the fact that two or more separate lamellar compositions having specific functions, e.g. cleansing and moisturizing the skin may be simultaneously dispensed in a partitionless container. A further advantage of using a lamellar composition is that elevated amounts of emollients may be added to the formula without affecting product stability. Unexpectedly, the lamellar phases in the inventive product remain separated (i.e. do not mix) at room temperature for at least 4 months and at high temperature (125° F) for at least two weeks.

20 "Stability" as used herein is therefore defined as the ability of the multiphase lamellar product to maintain the separation of each phase from the other under the above combinations of time and temperature.

In accordance with these and other aspects of the invention,  
25 the invention will now be described with reference to the  
accompanying drawing.

- 9 -

BRIEF DESCRIPTION OF FIGURE

Figure 1 is a perspective view showing one embodiment of the present invention wherein two separate liquid cleansing compositions in lamellar phase are contained in a partitionless container.

Referring to Figure 1 in more detail, a partitionless container 10 contains two separate liquid cleansing compositions 18 and 20 separated by a boundary layer 22. Also depicted in Figure 1 is a pump mechanism 12 having a single outlet 14 and a suction tube 16 with a single intake hole 24. Preferably, the cleansing compositions 18 and 20 are visually distinct signaling duality to the consumer. More preferably, the cleansing compositions 18 and 20 have different colors or distinctive visual clarity. In another embodiment of the invention, the partitionless container may contain more than two separate liquid cleaning compositions in the lamellar phase.

The present invention also relates to multiphase liquid lamellar cleansing compositions, wherein the liquid is in a lamellar phase, comprising a surfactant system, preferably a system which contains at least about 5 wt. %, preferably at least about 10 wt. % of surface active compounds. The inventive composition also includes an amphoteric and/or zwitterionic surfactant. Preferably the amphoteric or zwitterionic surfactant, or a blend thereof, is present at about 3 to 40 wt. %, more preferably at about 5 to 20 wt. %. The inventive composition also contains at least one anionic surfactant. Preferably the anionic surfactant is present at

- 10 -

about 3 to 40 wt. %, more preferably at about 5 to 20 wt. %. The inventive composition also contains a lamellar structurant. Preferably the lamellar structurant is present at about 0.3 to 15 wt. %, more preferably at about 0.5 to 5  
5 wt. %.

Each of the component phases of the inventive multiphase lamellar composition has a low shear viscosity in the range of about 80,000 to 300,000 centipoises (cps) measured at 0.5  
10 RPM using a T-bar spindle A according to the procedure described below. Preferably the viscosity ranges from about 100,000 to 200,000 cps and the difference in viscosity between abutting phases is in the range of 0 to 10 % expressed as a relative value.  
15

#### Surfactants

The surfactant system of the subject invention comprises 5 to 60% by weight, preferably 10 to 30% by wt. of the  
20 composition and comprises:

- (a) at least one anionic surfactant;
- (b) at least one amphoteric and/or zwitterionic surfactant;
- (c) at least one lamellar structurant compound; and
- 25 (d) optionally one or more nonionic surfactants, cationic surfactants, or blends thereof.

The anionic surfactant (which may comprise 3 to 40 % by wt. of total composition) may be, for example, an aliphatic  
30 sulfonate, such as a primary alkane (e.g., C<sub>8</sub>-C<sub>22</sub>) sulfonate,

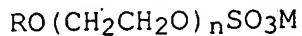
- 11 -

primary alkane (e.g., C<sub>8</sub>-C<sub>22</sub>) disulfonate, C<sub>8</sub>-C<sub>22</sub> alkene sulfonate, C<sub>8</sub>-C<sub>22</sub> hydroxyalkane sulfonate or alkyl glyceryl ether sulfonate (AGS); or an aromatic sulfonate such as alkyl benzene sulfonate, and the like.

5

The anionic may also be an alkyl sulfate (e.g., C<sub>12</sub>-C<sub>18</sub> alkyl sulfate) or alkyl ether sulfate (including alkyl glyceryl ether sulfates), and the like. Among the alkyl ether sulfates are those having the formula:

10

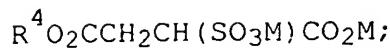


wherein R is an alkyl or alkenyl having 8 to 18 carbons, preferably 12 to 18 carbons, n has an average value of 15 greater than 1.0, preferably between 2 and 3; and M is a solubilizing cation such as sodium, potassium, ammonium or substituted ammonium. Ammonium and sodium lauryl ether sulfates are preferred.

20 The anionic may also be selected from alkyl sulfosuccinates (including mono- and dialkyl, e.g., C<sub>6</sub>-C<sub>22</sub> sulfosuccinates); alkyl and acyl taurates, alkyl and acyl sarcosinates, sulfoacetates, C<sub>8</sub>-C<sub>22</sub> alkyl phosphates and phosphates, alkyl phosphate esters and alkoxy alkyl phosphate esters, acyl 25 lactates, C<sub>8</sub>-C<sub>22</sub> monoalkyl succinates and maleates, sulphoacetates, and acyl isethionates, and the like.

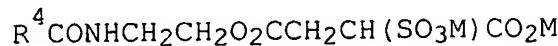
Sulfosuccinates may be monoalkyl sulfosuccinates having the formula:

- 12 -



amido-MEA sulfosuccinates of the formula

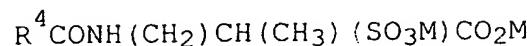
5



wherein  $R^4$  ranges from C<sub>8</sub>-C<sub>22</sub> alkyl and M is a solubilizing cation;

10

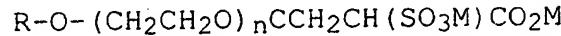
amido-MIPA sulfosuccinates of the formula



15 wherein  $R^4$  and M are as defined above.

Also included are alkoxylated citrate sulfosuccinates; and alkoxylated sulfosuccinates such as the following:

20



wherein n = 1 to 20; and M is as defined above.

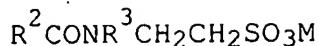
25

Sarcosinates are generally indicated by the formula RCON(CH<sub>3</sub>)CH<sub>2</sub>CO<sub>2</sub>M, wherein R ranges from C<sub>8</sub> to C<sub>20</sub> alkyl and M is a solubilizing cation.

Taurates are generally identified by the formula

30

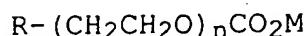
- 13 -



wherein  $R^2$  ranges from C<sub>8</sub>-C<sub>20</sub> alkyl,  $R^3$  ranges from C<sub>1</sub>-C<sub>4</sub> alkyl and M is a solubilizing cation.

5

Another class of anionics are carboxylates such as follows:



10 wherein R is C<sub>8</sub> to C<sub>20</sub> alkyl; n is 0 to 20; and M is as defined above.

Other suitable carboxylate are amido alkyl polypeptide carboxylates such as, for example, Monteine LCQ<sup>(R)</sup> by Seppic.

15

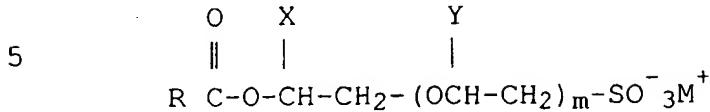
Other surfactants which may be used are the C<sub>8</sub>-C<sub>18</sub> acyl isethionates. These esters are prepared by reaction between alkali metal isethionate with mixed aliphatic fatty acids having from 6 to 18 carbon atoms and an iodine value of less 20 than 20. At least 75% of the mixed fatty acids have from 12 to 18 carbon atoms and up to 25% have from 6 to 10 carbon atoms.

Acyl isethionates, when present, will generally range from 25 about 0.5-15% by weight of the total composition. Preferably, this component is present from about 1 to about 10% by weight of the total composition.

The acyl isethionate may be an alkoxylated isethionate such 30 as is described in U.S. Patent No. 5,393,466, to Ilardi et

- 14 -

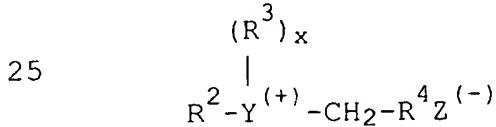
al., hereby incorporated by reference. This compound has the general formula:



wherein R is an alkyl group having 8 to 18 carbons, m is an integer from 1 to 4, X and Y are hydrogen or an alkyl group having 1 to 4 carbons and M<sup>+</sup> is a monovalent cation such as, for example, sodium, potassium or ammonium.

#### Zwitterionic and Amphoteric Surfactants

15 Zwitterionic surfactants are exemplified by those which may be broadly described as derivatives of aliphatic quaternary ammonium, phosphonium, and sulfonium compounds, in which the aliphatic radicals can be straight or branched chain, and wherein one of the aliphatic substituents contains from 20 about 8 to about 18 carbon atoms and one contains an anionic group, e.g., carboxy, sulfonate, sulfate, phosphate, or phosphonate. A general formula for these compounds is:



wherein R<sup>2</sup> is an alkyl, alkenyl, or hydroxy alkyl radical containing from about 8 to about 18 carbon atoms, from 0 to 30 about 10 ethylene oxide moieties and from 0 to about 1 glyceryl moiety; Y is selected from nitrogen, phosphorus, and sulfur atoms; R<sup>3</sup> is an alkyl or monohydroxyalkyl group

- 15 -

containing about 1 to about 3 carbon atoms; X is 1 when Y is a sulfur atom, and 2 when Y is a nitrogen or phosphorus atom; R<sup>4</sup> is an alkylene or hydroxyalkylene of from about 1 to about 4 carbon atoms and Z is a radical selected from 5 carboxylate, sulfonate, sulfate, phosphonate, and phosphate groups.

Examples of such surfactants include:

4-[N,N-di(2-hydroxyethyl)-N-octadecylammonio]-butane-1-carboxylate;

5-[S-3-hydroxypropyl-S-hexadecylsulfonio]-3-hydroxypentane-1-sulfate;

3-[P,P-diethyl-P-3,6,9-trioxatetradexocylphosphonio]-2-hydroxypropane-1-phosphate;

15 3-[N,N-dipropyl-N-3-dodecoxy-2-hydroxypropylammonio]-propane-1-phosphonate;

3-(N,N-dimethyl-N-hexadecylammonio)propane-1-sulfonate;

3-(N,N-dimethyl-N-hexadecylammonio)-2-hydroxypropane-1-sulfonate;

20 4-[N,N-di-(2-hydroxyethyl)-N-(2-hydroxydodecyl)ammonio]-butane-1-carboxylate;

3-[S-ethyl-S-(3-dodecoxy-2-hydroxypropyl)sulfonio]-propane-1-phosphate;

3-[P,P-dimethyl-P-dodecylphosphonio]-propane-1-phosphonate;

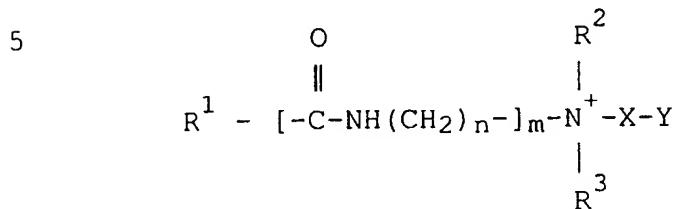
25 and

5-[N,N-di(3-hydroxypropyl)-N-hexadecylammonio]-2-hydroxy-pentane-1-sulfate.

Amphoteric detergents which are suitable for the present 30 invention include at least one acid group. This may be a carboxylic or a sulphonic acid group. They include

- 16 -

quaternary nitrogen and therefore are quaternary amido acids. They should generally include an alkyl or alkenyl group of 7 to 18 carbon atoms and usually comply with an overall structural formula:



10 wherein R<sup>1</sup> is an alkyl or alkenyl group having 7 to 18 carbon atoms;

R<sup>2</sup> and R<sup>3</sup> are each independently alkyl, hydroxyalkyl or a carboxyalkyl groups having 1 to 3 carbon atoms;

15

n is 2 to 4;

m is 0 to 1;

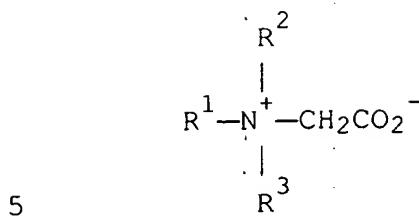
20 X is an alkylene group having 1 to 3 carbon atoms optionally substituted with hydroxyl, and

Y is -CO<sub>2</sub><sup>-</sup> or -SO<sub>3</sub><sup>-</sup>

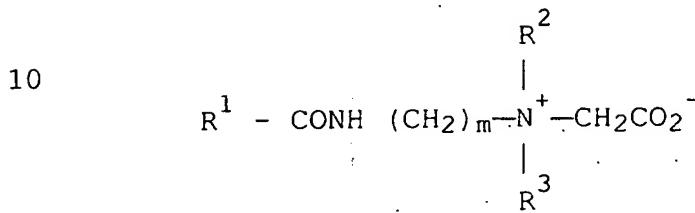
25 Suitable amphoteric detergents within the above general formula include simple betaines of formula:

30

- 17 -



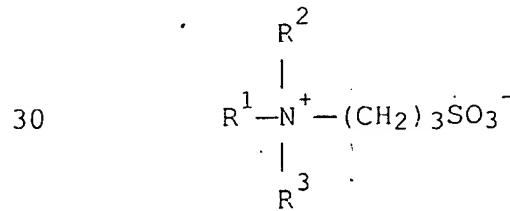
and amido betaines of formula:



15 wherein m is 2 or 3.

In both formulae  $\text{R}^1$ ,  $\text{R}^2$  and  $\text{R}^3$  are as defined previously.  $\text{R}^1$  may, in particular, be a mixture of C<sub>12</sub> and C<sub>14</sub> alkyl groups 20 derived from coconut so that at least half, preferably at least three quarters of the groups  $\text{R}^1$  have 10 to 14 carbon atoms.  $\text{R}^2$  and  $\text{R}^3$  are preferably methyl. A suitable betaine is cocoamidopropyl betaine.

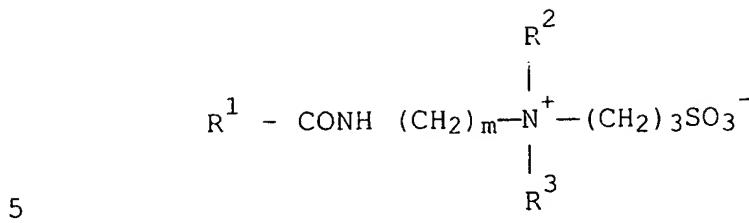
25 A further possibility is that the amphoteric detergent is a sulphobetaine of formula



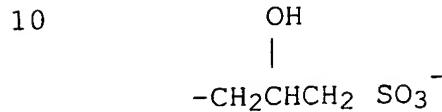
or

35

- 18 -



where m is 2 or 3, or variants of these in which  $-(CH_2)_3 SO_3^-$  is replaced by



In these formulae  $R^1$ ,  $R^2$  and  $R^3$  are as discussed previously.

15

Amphoacetates and diamphoacetates are also intended to be covered in possible zwitterionic and/or amphoteric compounds which may be used, especially C8 - C20 amphoacetates or mixtures thereof, and the like. A suitable amphoacetate is 20 sodium laurylamphoacetate.

The amphoteric/zwitterionic surfactant, when used, generally comprises 3 to 30%, preferably 5 to 20% by weight, more preferably 10 to 20% by weight of the composition.

25

A preferred surfactant system of the invention comprises the following:  
 anionic surfactant (e.g. alkali metal alkyl ethersulfate), 2-50%; amphoteric surfactant (e.g. alkyl betaine or alkyl 30 amphoacetate), 3-20%.

- 19 -

The surfactant system may also optionally comprise a nonionic surfactant.

Suitable nonionic surfactants include, in particular, the 5 reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide either alone or with propylene oxide. Specific nonionic detergent compounds are 10 alkyl (C<sub>6</sub>-C<sub>22</sub>) phenols-ethylene oxide condensates, the condensation products of aliphatic (C<sub>8</sub>-C<sub>18</sub>) primary or secondary, linear or branched alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylenediamine. 15 Other so-called nonionic detergent compounds include long chain tertiary amine oxides, long chain tertiary phosphine oxides and dialkyl sulphoxides, and the like.

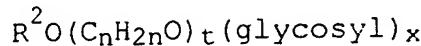
The nonionic may also be a sugar amide, such as a 20 polysaccharide amide. Specifically, the surfactant may be one of the lactobionamides described in U.S. Patent No. 5,389,279 to Au et al. (incorporated herein by reference) or it may be one of the sugar amides described in Patent No. 5,009,814 to Kelkenberg, hereby incorporated into the 25 subject application by reference.

Other surfactants which may be used are described in U.S. Patent No. 3,723,325 to Parran Jr. and alkyl polysaccharide nonionic surfactants as disclosed in U.S. Patent No.

- 20 -

4,565,647 to Llenado, both of which are also incorporated into the subject application by reference.

Preferred alkyl polysaccharides are alkylpolyglycosides of  
5 the formula



wherein  $R^2$  is selected from alkyl, alkylphenyl, hydroxyalkyl, hydroxyalkylphenyl, and mixtures thereof in which the alkyl groups contain from about 10 to about 18, 10 preferably from about 12 to about 14, carbon atoms; n is 0 to 3, preferably 2; t is from 0 to about 10, preferably 0; and x is from 1.3 to about 10, preferably from 1.3 to about 2.7. The glycosyl is preferably derived from glucose.

15 To prepare these compounds, the alcohol or alkylpolyethoxy alcohol is formed first and then reacted with glucose, or a source of glucose, to form the glucoside (attachment at the 1-position). The additional glycosyl units can then be attached between their 1-position and the preceding glycosyl 20 units 2-, 3-, 4- and/or 6-position, preferably predominantly the 2-position.

The nonionic surfactant generally comprises 3 to 40% by wt. of the composition, preferably 0 to 10% by wt. of the 25 composition.

Lamellar Structurant

The compositions of the invention utilize about 0.3% to 15% 30 by wt., preferably 0.5 to 10% by wt., more preferably 0.5 to

- 21 -

5% by wt. of a structuring agent in each phase which functions in the compositions to form a lamellar phase. The lamellar phase enables the compositions to suspend particles more readily (e.g., emollient particles) while still 5 maintaining good shear thinning properties. The lamellar phase also provides consumers with desired rheology ("heaping").

The structurant is preferably a fatty acid or ester 10 derivative thereof, a fatty alcohol, or trihydroxystearin, and the like. More preferably the structurant is selected from lauric or isostearic acid, or trihydroxystearin.

Suitable examples of fatty acids include C<sub>10</sub>-C<sub>22</sub> acids such as 15 the following: lauric acid, oleic acid, isostearic acid, linoleic acid; linolenic acid, ricinoleic acid, elaidic acid, arachidonic acid, myristoleic acid and palmitoleic acid, and the like. Ester derivatives include propylene glycol isostearate, propylene glycol oleate, glycetyl 20 isostearate, glycetyl oleate and polyglyceryl diisostearate, and the like.

#### Oil/Emollient

25 One of the principle benefits of the invention is the ability to suspend oil/emollient particles in one or more lamellar phases in the multiphase composition. The following oil/emollients may optionally be suspended in the compositions of the invention.

- 22 -

Various classes of oils are set forth below:

Vegetable oils: Arachis oil, castor oil, cocoa butter,  
5 coconut oil, corn oil, cotton seed oil, olive oil, palm  
kernel oil, rapeseed oil, safflower seed oil, sesame seed  
oil and soybean oil, and the like.

Esters: Butyl myristate, cetyl palmitate, decyloleate,  
10 glyceryl laurate, glyceryl ricinoleate, glyceryl stearate,  
glyceryl isostearate, hexyl laurate, isobutyl palmitate,  
isocetyl stearate, isopropyl isostearate, isopropyl laurate,  
isopropyl linoleate, isopropyl myristate, isopropyl  
palmitate, isopropyl stearate, propylene glycol monolaurate,  
15 propylene glycol ricinoleate, propylene glycol stearate, and  
propylene glycol isostearate, and the like.

Animal Fats: acetylated lanolin alcohols, lanolin, lard,  
mink oil and tallow, and the like.

20 Other examples of oil/emollients include mineral oil,  
petrolatum, silicone oil such as dimethyl polysiloxane,  
lauryl and myristyl lactate, fatty acid oils, triglycerides,  
glycerin, and the like.

25 The emollient/oil is generally used in an amount from about  
0 to 70%, preferably 5 to 40% by wt. of the phase in which  
it is to be found. Generally, it should comprise no more  
than 70% of such phase. A portion of the emollient may be  
30 present in the form of solid or semi-solid beads. The beads

- 23 -

are used in an amount from about 0 to 10%, preferably 0 to 5% of the phase.

In addition, the multiphase lamellar compositions of the  
5 invention may include optional ingredients as follows:

Organic solvents, such as ethanol; auxiliary thickeners,  
sequestering agents, such as tetrasodium  
10 ethylenediaminetetraacetate (EDTA), EHDP or mixtures in an  
amount of 0.01 to 1%, preferably 0.01 to 0.05%; and coloring  
agents, opacifiers and pearlizers such as zinc stearate,  
magnesium stearate, TiO<sub>2</sub>, EGMS (ethylene glycol monostearate)  
or Lytron 621 (Styrene/Acrylate copolymer); all of which are  
useful in enhancing the appearance or cosmetic properties of  
15 the product.

The compositions may further comprise antimicrobials such as  
2-hydroxy-4,2'4'-trichlorodiphenylether(DP3000);  
preservatives such as dimethyloldimethylhydantoin (Glydant  
20 XL1000), parabens, sorbic acid etc.

The compositions may also comprise coconut acyl mono- or  
diethanol amides and the like as suds boosters.

25 Antioxidants such as, for example, butylated hydroxytoluene  
(BHT) may be used advantageously in amounts of about 0.01%  
or higher if appropriate.

Cationic conditioners which may be used include Quatrisoft  
30 LM-200 Polyquaternium-24, Merquat Plus 3330 - Polyquaternium  
39; and Jaguar<sup>(R)</sup> type conditioners.

- 24 -

Another optional ingredient which may be added are the deflocculating polymers such as are taught in U.S. Patent No. 5,147,576 to Montague, incorporated herein by reference.

5

Other ingredients which may be included are exfoliants such as polyoxyethylene beads, walnut sheets and apricot seeds, and the like. PH and viscosity adjusters may be used such as citric acid, glycolic acid, lactic acid, other alpha or 10 beta hydroxy acids, and the like.

The multiphase compositions of the invention, as noted, are lamellar compositions. In particular, the lamellar phase comprises 20 to 80%, preferably 30 to 65% of the total phase volume of each phase. The phase volume may be measured, for example, by conductivity measurements or other measurements which are well known to those skilled in the art. While not wishing to be bound by theory, higher phase volume is believed to provide better suspension of emollients.

20

The invention will now be described in greater detail by way of the following non-limiting examples. The examples are for illustrative purposes only and not intended to limit the invention in any way.

25

Except in the examples, or where otherwise explicitly indicated, all numbers in this description indicating amounts or ratios of materials or conditions or reaction, physical properties of materials and/or use are to be understood as modified by the word "about".

- 25 -

Where used in the specification, the term "comprising" is intended to include the presence of stated features, integers, steps, components, but not to preclude the presence or addition of one or more features, integers,  
5 steps, components or groups thereof.

All percentages in the specification and examples are intended to be by weight unless stated otherwise.

10 Examples 1 - 4 are inventive compositions having two lamellar phases, denoted stripe A and stripe B. All the compositions were found to remain separated (i.e. did not mix) at room temperature for at least 4 months and at high temperature (125° F) for at least two weeks. The  
15 compositions were held in a transparent PET container as depicted in Figure 1. The dispensed product in all cases was found to contain each stripe in the range of 1 - 99 weight %.

20

25

30

- 26 -

Example 1:

| COMPONENT                                      | STRIPES A<br>% IN FORMULATION |
|--|-------------------------------|
| SODIUM LAUROAMPHOACETATE                       | 7                             |
| SODIUM LAURETH SULFATE                         | 14                            |
| CETYL ACETATE AND ACETYLATED LANOLIN ALCOHOL   | 0.5                           |
| LAURIC ACID                                    | 2.5-3.0*                      |
| SUNFLOWER SEED OIL                             | 3                             |
| COCAMIDE MEA                                   | 2                             |
| GLYCERIN                                       | 2                             |
| GUAR HYDROXYPROPYL TRIMONIUM CHLORIDE          | 0.5                           |
| CITRIC ACID                                    | 1.2                           |
| TITANIUM DIOXIDE                               | 0.2                           |
| DMDM HYDANTOIN/<br>IODOPROPYNYL BUTYLCARBAMATE | 0.2                           |
| EDTA   | 0.02                          |
| EHDP (Etidronic Acid)                          | 0.02                          |
| PERFUME  | 0.5                           |
| WATER  | TO 100.0                      |

\* to adjust viscosities

- 27 -

| Stripe B<br>COMPONENT                          | % IN FORMULATION |
|--|------------------|
| SODIUM LAUROAMPHOACETATE                       | 3                |
| SODIUM LAURETH SULFATE                         | 6                |
| CETYL ACETATE AND ACETYLATED LANOLIN ALCOHOL   | 1.5              |
| LAURIC ACID                                    | 2.5-3.2*         |
| SUNFLOWER SEED OIL                             | 8                |
| COCAMIDE MEA                                   | 2                |
| GLYCERIN                                       | 2                |
| GUAR HYDROXYPROPYL TRIMONIUM CHLORIDE          | 0.5              |
| CITRIC ACID                                    | 0.7              |
| RED DYE SOLUTION @ 0.1%                        | 0.7              |
| DMDM HYDANTOIN/<br>IODOPROPYNYL BUTYLCARBAMATE | 0.2              |
| VITAMIN E ACETATE                              | 0.1              |
| EDTA   | 0.02             |
| EHDP (Etidronic Acid)                          | 0.02             |
| PERFUME  | 0.5              |
| WATER  | TO 100.0         |

\* to adjust viscosities

- 28 -

## Example 2:

## Stripe A

| COMPONENT                                      | % IN FORMULATION |
|--|------------------|
| SODIUM LAUROAMPHOACETATE                       | 10               |
| SODIUM LAURETH SULFATE                         | 15               |
| BEADS  | 1                |
| LAURIC ACID                                    | 1.2              |
| SUNFLOWER SEED OIL                             | 10               |
| COCAMIDE MEA                                   | 2.5              |
| GUAR HYDROXYPROPYL TRIMONIUM CHLORIDE          | 0.5              |
| CITRIC ACID                                    | 0.5              |
| TITANIUM DIOXIDE                               | 0.2              |
| DMDM HYDANTOIN/<br>IODOPROPYNYL BUTYLCARBAMATE | 0.2              |
| EDTA   | 0.02             |
| EHDP (Etidronic Acid)                          | 0.02             |
| PERFUME  | 1                |
| WATER  | TO 100.0         |

\* to adjust viscosities

5

## Stripe B

| COMPONENT                                      | % IN FORMULATION |
|--|------------------|
| SODIUM LAUROAMPHOACETATE                       | 18               |
| SODIUM LAURETH SULFATE                         | 5                |
| LAURIC ACID                                    | 1.6              |
| SUNFLOWER SEED OIL                             | 10               |
| TRIHYDROXYSTEARIN                              | 0.5              |
| COCAMIDE MEA                                   | 2.5              |
| GLYCERIN                                       | 2                |
| GUAR HYDROXYPROPYL TRIMONIUM CHLORIDE          | 0.5              |
| CITRIC ACID                                    | 1                |
| RED DYE SOLUTION @ 0.1%                        | 0.1              |
| DMDM HYDANTOIN/<br>IODOPROPYNYL BUTYLCARBAMATE | 0.2              |
| VITAMIN E ACETATE                              | 0.2              |
| EDTA   | 0.02             |
| EHDP (Etidronic Acid)                          | 0.02             |
| PERFUME  | 1.5              |
| WATER  | TO 100.0         |

• to adjust viscosities

- 29 -

Example 3:

Stripe A

| COMPONENT                                    | % IN FORMULATION |
|--|------------------|
| SODIUM LAUROAMPHOACETATE                     | 5                |
| SODIUM LAURETH SULFATE                       | 15               |
| SODIUM LAUROYL SARCOSINATE                   | 4                |
| CETYL ACETATE AND ACETYLATED LANOLIN ALCOHOL | 0.5              |
| LAURIC ACID                                  | 3.6              |
| COCAMIDE MEA                                 | 2                |
| GLYCERIN                                     | 4                |
| GUAR HYDROXYPROPYL TRIMONIUM CHLORIDE        | 0.5              |
| CITRIC ACID                                  | 0.7              |
| TITANIUM DIOXIDE                             | 0.2              |
| DMDM HYDANTOIN/IODOPROPYNYL BUTYLCARBAMATE   | 0.2              |
| EDTA   | 0.02             |
| EHDP (Etidronic Acid)                        | 0.02             |
| PERFUME                                      | 0.5              |
| WATER  | TO 100.0         |

\* to adjust viscosities

Stripe B

| COMPONENT                                    | % IN FORMULATION |
|--|------------------|
| SODIUM LAUROAMPHOACETATE                     | 12               |
| SODIUM LAURETH SULFATE                       | 9                |
| CETYL ACETATE AND ACETYLATED LANOLIN ALCOHOL | 1                |
| LAURIC ACID                                  | 3.4              |
| SUNFLOWER SEED OIL                           | 10               |
| COCAMIDE MEA                                 | 2                |
| GLYCERIN                                     | 6                |
| GUAR HYDROXYPROPYL TRIMONIUM CHLORIDE        | 1                |
| CITRIC ACID                                  | 0.8              |
| BLUE DYE SOLUTION @ 0.1%                     | 0.05             |
| DMDM HYDANTOIN/IODOPROPYNYL BUTYLCARBAMATE   | 0.2              |
| VITAMIN A PALMITATE                          | 0.2              |
| EDTA   | 0.02             |
| EHDP (Etidronic Acid)                        | 0.02             |
| PERFUME                                      | 1                |
| WATER  | TO 100.0         |

\* to adjust viscosities

- 30 -

## Example 4:

## Stripe A

| COMPONENT                                      | % IN FORMULATION |
|--|------------------|
| SODIUM LAUROAMPHOACETATE                       | 10               |
| SODIUM LAURETH SULFATE                         | 7                |
| SODIUM LAURYL SULFATE                          | 3                |
| ISOSTEARIC ACID                                | 3.5              |
| SUNFLOWER SEED OIL                             | 5                |
| COCAMIDE MEA                                   | 2                |
| GLYCERIN                                       | 7                |
| GUAR HYDROXYPROPYL TRIMONIUM CHLORIDE          | 0.6              |
| CITRIC ACID                                    | 0.9              |
| TITANIUM DIOXIDE                               | 0.3              |
| DMDM HYDANTOIN/<br>IODOPROPYNYL BUTYLCARBAMATE | 0.2              |
| EDTA   | 0.02             |
| EHDP (Etidronic Acid)                          | 0.02             |
| TRICLOSAN                                      | 0.5              |
| PERFUME  | 0.6              |
| WATER  | TO 100.0         |

\* to adjust viscosities  
 Stripe B

| COMPONENT                                      | % IN FORMULATION |
|--|------------------|
| SODIUM LAUROAMPHOACETATE                       | 4                |
| AMMONIUM LAURETH SULFATE                       | 3                |
| AMMONIUM LAURYL SULFATE                        | 4                |
| LAURIC ACID                                    | 3.2              |
| PETROLATUM                                     | 15               |
| COCAMIDE MEA                                   | 2                |
| GLYCERIN                                       | 8                |
| GUAR HYDROXYPROPYL TRIMONIUM CHLORIDE          | 1                |
| CITRIC ACID                                    | 0.9              |
| RED DYE SOLUTION @ 0.1%                        | 0.1              |
| DMDM HYDANTOIN/<br>IODOPROPYNYL BUTYLCARBAMATE | 0.2              |
| VITAMIN E ACETATE                              | 0.2              |
| EDTA   | 0.02             |
| EHDP (Etidronic Acid)                          | 0.02             |
| PERFUME  | 1.4              |
| WATER  | TO 100.0         |

• to adjust viscosities

- 31 -

Viscosity measurements were obtained in accordance with the following protocol:

Viscosity Measurement

5    Scope:

This method covers the measurement of the viscosity of the finished product.

Apparatus:

10

Brookfield RVT Viscometer with Helipath Accessory;  
Chuck, weight and closer assembly for T-bar attachment;  
T-bar Spindle A;  
Plastic cups diameter greater than 2.5 inches.

15

Procedure:

1. Verify that the viscometer and the helipath stand are level by referring to the bubble levels on the back of  
20 the instrument.
2. Connect the chuck/closer/weight assembly to the Viscometer (Note the left-hand coupling threads).
- 25 3. Clean Spindle A with deionized water and pat dry with a Kimwipe sheet. Slide the spindle in the closer and tighten.
4. Set the rotational speed at 0.5 RPM. In case of a  
30 digital viscometer (DV) select the % mode and press autozero with the motor switch on.

- 32 -

5. Place the product in a plastic cup with inner diameter of greater than 2.5 inches. The height of the product in the cup should be at least 3 inches. The temperature of the product should be 25°C.
10. Lower the spindle into the product (~1/4 inches). Set the adjustable stops of the helipath stand so that the spindle does not touch the bottom of the plastic cup or come out of the sample.
15. Start the viscometer and allow the dial to make one or two revolutions before turning on the Helipath stand. Note the dial reading as the helipath stand passes the middle of its downward traverse.
20. Multiply the dial reading by a factor of 4,000 and report the viscosity reading in cps.

While this invention has been described with respect to particular embodiments thereof, it is apparent that numerous other forms and modifications of the invention will be obvious to those skilled in the art.

- 33 -

**CLAIMS**

1. A stable, extrudable, multiphase aqueous lamellar structured liquid cleansing composition, comprising:

5

at least two aqueous lamellar structured phases which abut each other;

10 a surfactant selected from amphoteric, zwitterionic, or mixtures thereof;

an anionic surfactant;

15 a lamellar structurant present in each lamellar phase of the liquid composition, selected from fatty acids, fatty esters, trihydroxystearin, fatty alcohols; and

wherein each of the lamellar phases has a minimum low shear viscosity value of 80 cps at 25°C.

20

2. A composition according to claim 1 wherein each lamellar phase has a maximum low shear viscosity of 300K cps at 25°C.

25 3. A composition according to claim 1 or claim 2 wherein two lamellar phases are present.

4. A composition according to any of the preceding claims wherein each of the lamellar phases has a low shear viscosity value of between 100K and 200K cps at 25°C.

30

- 34 -

5. A composition according to any of the preceding claims wherein the amphoteric surfactant concentration in each phase is in the range of about 3 to 30 weight percent.
- 5 6. A composition according to any of the preceding claims wherein the anionic surfactant concentration in each phase is in the range of about 3 to 40 weight percent.
- 10 7. A composition according to any of the preceding claims wherein the nonionic surfactant concentration in each phase is in the range of about 3 to 40 weight percent.
- 15 8. A composition according to any of the preceding claims wherein the emollient concentration in each phase is in the range of about 0 to 70 weight percent.
9. A composition according to any of the preceding claims wherein the lamellar structurant concentration in each phase is in the range of about 0.3 to 15 weight percent.
- 20 10. A composition according to any of the preceding claims wherein the amphoteric or zwitterionic surfactant in each phase is selected from either cocamidopropyl betaine, or an alkali metal salt of alkyl amboacetate.
- 25 11. A composition according to any of the preceding claims wherein the anionic surfactant is selected from alkali metal or ammonium alkyl ether sulfate, alkali metal or ammonium alkyl sarcosinate, alkali metal or ammonium alkyl sulfosuccinate, and alkali metal or ammonium alkyl sulfate.

- 35 -

12. A composition according to any of the preceding claims wherein mixing of each abutting lamellar phase is prevented across the interfacial boundary surfaces of the abutting phases, when the compositions are stored at 5  $25^{\circ}\text{C}$  for at least 120 days or at least  $50^{\circ}\text{C}$  for 14 days.

13. A composition according to any of the preceding claims wherein the lamellar structurant is selected from lauric acid, isostearic acid, trihydroxystearin, palm kernel acid, capric acid, oleic acid, and caprylic acid. 10

14. An extrudable aqueous multiphase lamellar structured liquid composition, comprising:

15 at least two abutting lamellar phases;

at least 5 weight percent of surface active material in each lamellar phase;

20 about 3 to 30 weight percent of one or more amphoteric, or zwitterionic surfactants or a blend thereof in each lamellar phase;

about 3 to 40 weight percent of an anionic surfactant in 25 each the lamellar phase;

about 0.5 to 10 weight percent of a lamellar structurant selected from fatty acids, fatty esters, fatty alcohols, or trihydroxystearin in each lamellar phase;

30 and

- 36 -

wherein each lamellar phase has a low shear viscosity value between 80 and 300 K cps at 25°C.

15. An extrudable multiphase aqueous lamellar structured liquid cleansing product contained in a partitionless container.
16. A product according to claim 15 wherein the container has a single opening for dispensing a multiphase lamellar composition.
17. A product according to claim 16 wherein the dispensed composition has at least 1 weight percent of each lamellar phase contained within the partitionless container.
18. A product according to claim 16 wherein the opening has a single pump attached thereto.
- 20 19. A product according to any of claims 15 to 18 wherein each lamellar phase has a distinct physical appearance.
- 25 20. A product according to claim 19 wherein each lamellar phase has a distinct color.
21. A product according to any of claims 15 to 20 wherein there are two lamellar phases.
- 30 22. A method of using an extrudable multiphase aqueous lamellar structured liquid cleansing composition, the composition having at least two abutting phases,

- 37 -

comprising:

a surfactant selected from amphoteric, zwitterionic, or mixtures thereof;

5

an anionic surfactant;

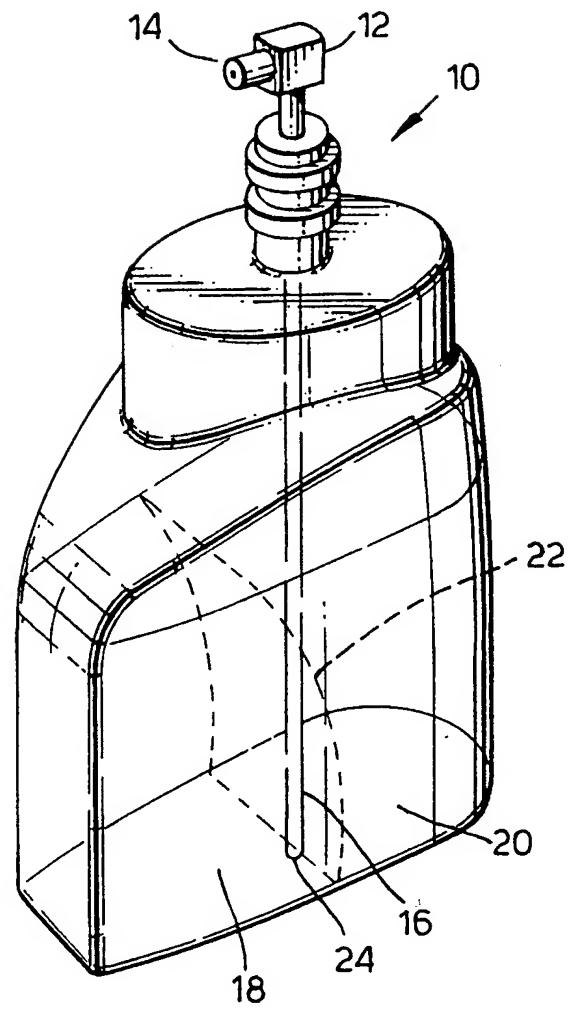
10

a lamellar structurant present in each phase of the liquid composition, selected from fatty acids, fatty esters, trihydroxystearin, fatty alcohols; and

wherein each of the lamellar phases has a low shear viscosity value between 80 and 300 K cps at 25° C.

15

Fig.1.



## INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 01/02458

|  |           |          |          |           |
|--|-----------|----------|----------|-----------|
| <b>A. CLASSIFICATION OF SUBJECT MATTER</b> |           |          |          |           |
| IPC 7                                      | C11D17/00 | C11D1/94 | C11D3/20 | C11D17/04 |
| A45D40/00                                  |           |          |          |           |

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C11D A61K A45D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, EPO-Internal, PAJ

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category | Citation of document, with indication, where appropriate, of the relevant passages  | Relevant to claim No.          |
|----------|---|--------------------------------|
| Y        | WO 98 24399 A (HENKEL KGAA)<br>11 June 1998 (1998-06-11)<br>cited in the application<br>claims 1,4; examples<br>---   | 1-17,<br>19-22                 |
| Y        | WO 97 05857 A (UNILEVER PLC ; UNILEVER NV (NL)) 20 February 1997 (1997-02-20)<br>cited in the application<br>page 6, line 30 -page 7, line 10<br>page 16, line 1 -page 17, line 9; claim 1<br>--- | 1-17,<br>19-22                 |
| A        | US 5 612 307 A (CHAMBERS JOHN G ET AL)<br>18 March 1997 (1997-03-18)<br>cited in the application<br>column 6, line 43 - line 53; examples<br>---  | 1-6,<br>10-12,<br>14,22<br>-/- |

 Further documents are listed in the continuation of box C. Patent family members are listed in annex.

## Special categories of cited documents:

- 'A' document defining the general state of the art which is not considered to be of particular relevance
- 'E' earlier document but published on or after the international filing date
- 'L' document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- 'O' document referring to an oral disclosure, use, exhibition or other means
- 'P' document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

- \*X\* document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

- \*Y\* document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

- \*8\* document member of the same patent family

Date of the actual completion of the international search

Date of mailing of the international search report

18 July 2001

24/07/2001

## Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
 NL - 2280 HV Rijswijk  
 Tel. (+31-70) 340-2040, Tx. 31 651 epo nl.  
 Fax: (+31-70) 340-3016

## Authorized officer

Saunders, T

## INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 01/02458

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

| Category | Citation of document, with indication, where appropriate, of the relevant passages   | Relevant to claim No. |
|----------|--|-----------------------|
| A        | WO 99 32069 A (UNILEVER PLC ; LEVER<br>HINDUSTAN LTD (IN); UNILEVER NV (NL))<br>1 July 1999 (1999-07-01)<br>page 19, line 13 -page 20, line 4; claims<br>1,10<br>----- | 1,2,<br>4-14,22       |

## INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 01/02458

| Patent document cited in search report |   | Publication date | Patent family member(s)   | Publication date   |
|--|---|------------------|---|--|
| WO 9824399                             | A | 11-06-1998       | DE 19650952 A<br>CA 2246316 A<br>CN 1214627 A<br>CZ 9802847 A<br>EP 0907345 A<br>HU 0000405 A<br>NO 983618 A<br>PL 328517 A<br>SK 120698 A  | 10-06-1998<br>11-06-1998<br>21-04-1999<br>17-02-1999<br>14-04-1999<br>28-08-2000<br>02-10-1998<br>01-02-1999<br>11-01-1999   |
| WO 9705857                             | A | 20-02-1997       | AU 719223 B<br>AU 6737396 A<br>BR 9609893 A<br>CA 2222274 A<br>CN 1192675 A<br>CZ 9800355 A<br>EP 0839023 A<br>HU 9901681 A<br>JP 11513053 T<br>PL 324889 A<br>US 6077816 A<br>US 5952286 A | 04-05-2000<br>05-03-1997<br>25-05-1999<br>20-02-1997<br>09-09-1998<br>13-05-1998<br>06-05-1998<br>29-11-1999<br>09-11-1999<br>22-06-1998<br>20-06-2000<br>14-09-1999 |
| US 5612307                             | A | 18-03-1997       | AU 2985895 A<br>BR 9508321 A<br>CA 2190738 A<br>EP 0755243 A<br>WO 9602230 A<br>JP 2977908 B<br>JP 9512826 T<br>KR 249451 B<br>ZA 9505972 A   | 16-02-1996<br>06-01-1998<br>01-02-1996<br>29-01-1997<br>01-02-1996<br>15-11-1999<br>22-12-1997<br>01-04-2000<br>20-01-1997   |
| WO 9932069                             | A | 01-07-1999       | US 6174846 B<br>AU 2271999 A<br>CN 1284857 T<br>EP 1037601 A<br>HU 0100099 A  | 16-01-2001<br>12-07-1999<br>21-02-2001<br>27-09-2000<br>28-05-2001   |

**THIS PAGE BLANK (USPTO)**